PATENT ABSTRACTS OF JAPAN

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(54) SEA URCHIN-LIKE ZINC OXIDE AND METHOD FOR PRODUCING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To firstly provide urchin-like zinc oxide which consists of a number of acicular particles and has large specific surface area and relatively high strength and to secondly provide a method for producing the urchin-like zinc oxide where the zinc oxides of other shapes are hardly mixed, at the low firing temperature and with a high yield in a production process, while there is no possibility of generating toxic gases.

SOLUTION: The zinc oxide has an urchin-like shape formed by aggregation of one ends of ≥10 acicular particles. In the method for manufacturing the urchin-like zinc oxide, the product obtained by mixing an aqueous solution (I) containing carbonate ions and/or bicarbonate ions and ammonium ions and an aqueous solution (II) containing zinc ions is fired at ≥ 300°C. The zinc ion concentration in the aqueous solution (II) is set lower than the total ion concentration of the carbonate ions and bicarbonate ions in the aqueous solution (I) and the pH of the aqueous solution (I) is regulated to 2 to 7.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]
This invention relates to the zinc oxide whose configuration is a sea urchin-like, and its manufacture approach.
[0002]
[Description of the Prior Art]
Zinc-oxide powder is used in fields, such as a pigment, drugs, a catalyst, or an electronic ingredient, and the zinc oxide of the shape of a scale, a globular shape, and needlelike ** is developed as the configuration. About a needlelike particle, the method of processing the aqueous acids of a zinc compound by a hexamethylenetetramine etc. is common as the manufacture approach.
[0003]
Recently, needlelike or the spherical zinc oxide which has a fibrous projection is also proposed from the front face of the core of a particle. For example, in JP,11-49516,A, the approach of compounding the zinc oxide which has a letter object of a projection on the surface of a spherical particle by calcinating an organic zinc compound at the temperature of 600-800 degrees C is indicated. As for the zinc oxide of such a configuration (the shape of confetti), the application to an ultraviolet ray absorbent, a conductive ingredient, catalyst support, etc. is expected.
[0004]
However, it is necessary to set temperature at the time of baking as this official report highly by the approach of a publication (600-800 degrees C). Moreover, since the organic zinc compound is used for a start raw material, a possibility that a toxic gas may occur is in a baking process. Furthermore, the yield of a confetti-like zinc oxide is as low as about ten%, and, moreover, it can obtain only under mixture with the zinc oxide of another configuration.
Moreover, since the spherical particle exists in the core, a limitation produces the zinc oxide obtained by doing in this way in specific surface area. Furthermore, if it does not make [many /. very] the addition to a binder in mixing such a zinc oxide to a binder and using it as a conductive ingredient, sufficient electric conduction effectiveness cannot be acquired.
[0005]
On the other hand, the so-called zinc oxide of the shape of the structure which is acquired by the vapor phase oxidation of a metal zinc steam and in which several needle crystal banded together, and a tetrapod is also known.
However, in order to make metal zinc into a steam, it is necessary to make an ambient atmosphere into an elevated temperature, therefore in composition of such a zinc oxide, special equipment is needed.
Moreover, in this zinc oxide, there is little needle crystal as four, reinforcement is weak and specific surface area also becomes small.
[0006]
[Patent reference 1] JP,11-49516,A (claim)
[0007]
[Problem(s) to be Solved by the Invention]
This invention is made in view of such a point.
The first technical problem in this invention consists of a needlelike particle of many numbers, and its specific surface area is large, and it is that reinforcement also offers a comparatively strong sea urchin-like zinc oxide.

In a manufacture process, burning temperature cannot be low, and a toxic gas cannot occur, and the second technical problem in this invention is still higher yield, and is offering the approach the zinc oxide of a sea urchin-like configuration being manufactured without hardly intermingling the zinc oxide of other configurations.

[0008]

[Means for Solving the Problem]

In order to solve the above-mentioned technical problem, this invention person resulted that the sea urchin-like zinc oxide of a new configuration was obtained by the specific manufacture approach in completion of a header and this invention wholeheartedly as a result of examination.

That is, this invention has the following descriptions.

The zinc oxide characterized by for one end of 1.10 or more needlelike particles gathering, and forming the sea urchin-like configuration.

2. It is the Manufacture Approach of Zinc Oxide of Calcinating Product Which Mixed Water Solution (I) Containing Carbonate Ion and/or Bicarbonate, and Ammonium Ion, and Water Solution (II) Containing Zinc Ion, and was Obtained above 300 Degrees C,

Rather than the sum total ion concentration of carbonate ion and the bicarbonate in a water solution (I), the zinc ion concentration in a water solution (II) is set up low,

pH of a water solution (II) is set to 2-7.

The manufacture approach of the sea urchin-like zinc oxide characterized by things.

3. Manufacture approach of sea urchin-like zinc oxide given in 2. characterized by making temperature of water solution (I) into 15-45 degrees C.

[0009]

[Embodiment of the Invention]

Hereafter, this invention is explained to a detail with the gestalt of the operation.

[0010]

In this invention, the water solution (I) containing carbonate ion and/or the bicarbonate, and ammonium ion and the water solution (II) containing zinc ion are mixed first.

[0011]

A water solution (I) contains carbonate ion and/or the bicarbonate, and ammonium ion. Although especially the generation source of these ion is not limited, one or more sorts chosen from an ammonium hydrogencarbonate and an ammonium carbonate are suitable.

As a generation source of the zinc ion in a water solution (II), a zinc chloride, a zinc sulfate, zinc nitrate, zinc acetate, etc. are mentioned, for example. Among these, in this invention, a zinc chloride is suitable.

[0012]

In this invention, the matter (henceforth a "sea urchin-like product") which consists of a hydroxyl ion, carbonate ion, and zinc ion generates by dropping a water solution (II) at a water solution (I) gradually. After a detailed nuclear material forms such a sea urchin-like product, balling-up is checked and it is considered that a needle shape crystal grows.

[0013]

In case a water solution (II) is dropped at a water solution (I), it is necessary to set up low the zinc ion concentration (only henceforth "concentration of a water solution (II)") in a water solution (II), and to set pH of a water solution (II) as 2-7 further rather than the sum density (only henceforth "concentration of a water solution (I)") of the carbonate ion in a water solution (I), and the bicarbonate. A sea urchin-like product can be obtained if it is under such a condition.

[0014]

Since rapid neutralization arises when the concentration of a water solution (II) is more than the concentration of a water solution (I), a sea urchin-like product cannot be obtained.

Suitable concentration conditions are that the concentration of the water solution (II) to the concentration of a water solution (I) is 60% or less (preferably 30% or less, still more preferably 15% or less).

[0015]

A sea urchin-like product cannot be obtained when pH of a water solution (II) is lower than 2. When pH is higher than 7, it becomes easy to produce precipitate in the water solution (II) itself. More suitable pH range is 2-6.

pH of a water solution (I) is usually six to about eight.
[0016]
It is desirable to set the temperature of a water solution (I) as 15-45 degrees C (further 20-45 degrees C) in this invention. With [the temperature of a water solution (I)] such within the limits, a sea urchin-like product can be obtained more certainly.
As for the temperature of a water solution (II), it is desirable to set up lower than the temperature of a water solution (I).
[0017]
Although especially the amount of mixing of the water solution (II) to a water solution (I) is not limited, it is more desirable than the total number of mols of carbonate ion and the bicarbonate to make it the total number of mols of zinc ion become small.
[0018]
If the sea urchin-like product generated by mixing with a water solution (I) and a water solution (II) is calcinated at the temperature of 300 degrees C or more, dehydration and a decarboxylation arise and a sea urchin-like zinc oxide can be obtained. Although burning temperature should just be 300 degrees C or more, it is 300 degrees C - 600 degrees C preferably.
What is necessary is not to need a special ambient atmosphere but just to carry out by the open system among atmospheric air about a baking process using usual baking equipment. When aiming at composition of the low suboxide, such as conductive grant, a baking phase can also be adjusted to a non-oxidizing atmosphere.
[0019]
The zinc oxide obtained by the above approach has a sea urchin-like configuration. One end of ten or more needlelike particles gathers, and, specifically, the sea urchin-like particle is formed. The particle diameter of a sea urchin-like particle is 0.5-60 micrometers in general. The die length of the needlelike particle which constitutes a sea urchin-like particle is about 0.25-30 micrometers, and a path is usually about 0.01-1 micrometer.
[0020]
The sea urchin-like zinc oxide of this invention can be used as an ultraviolet ray absorbent, a conductive ingredient, catalyst support, a reinforcing agent, a bulking agent, a pigment, a vulcanization promotion assistant, etc. As a field of the invention, a coating, ink, paints, glass, glaze, plating, water repellent, dentistry cement, a gas sensor, drugs, petroleum refining, a cell, an electrophotography ingredient, a fluorescent substance, an electromagnetic wave absorber, acoustic material, etc. are mentioned.
Since specific surface area becomes large because especially this invention zinc oxide becomes sea urchin-like, the remarkable improvement in the engine performance is predicted depending on the application used. For example, since the contact probability of sea urchin-like zinc oxides becomes high when it compounds with resin etc. and this invention zinc oxide is used for a conductive ingredient, compared with a spherical zinc oxide etc., little and sufficient electric conduction effectiveness is expectable.
In addition, when using this invention zinc oxide for the various above applications, various processings can also be performed to extent which does not spoil a sea urchin configuration.
[0021]
[Example]
An example is shown below and this invention is explained more to a detail.
[0022]
(Example 1)
200ml of zinc chloride water solutions of 0.2 mol/l and pH3 was gradually dropped to 400ml of ammonium-hydrogencarbonate water solutions of 1.5 mol/l. In addition, temperature of an ammonium-hydrogencarbonate water solution was made into 20 degrees C.
Thus, the zinc oxide with a particle size of 17-20 micrometers was obtained by washing and filtering the obtained product and calcinating at 400 more degrees C.
The configuration of the obtained zinc oxide was observed using the scanning electron microscope (JEOL [Co., Ltd.] make: JSM5301LV). The result is shown in drawing 1 . Having been the sea urchin-like particle to which one end of many needlelike particles gathered to about one point was admitted so that more clearly than drawing.
Subsequently, the specific surface area of the obtained zinc oxide was used as gas-hold-up measurement gas

with P-surface area measuring device 700 mold (the Shibata science device industrial incorporated company make), nitrogen was used as helium and adsorption gas, and it measured by the BET multipoint method. The specific surface area of the zinc oxide obtained in the example 1 was 35m2/g.

[0023]

(Example 2)

200ml of zinc chloride water solutions of 0.2 mol/l and pH3 was gradually dropped to 400ml of ammonium-hydrogencarbonate water solutions of 1.5 mol/l. In addition, temperature of an ammonium-hydrogencarbonate water solution was made into 40 degrees C.

Thus, the zinc oxide with a particle size of 15-27 micrometers was obtained by washing and filtering the obtained product and calcinating at 400 more degrees C.

In the example 2, the sea urchin-like particle shown in drawing 2 was obtained. The specific surface area of this zinc oxide was 50m2/g.

[0024]

(Example 3)

200ml of zinc chloride water solutions of 0.2 mol/l and pH3 was gradually dropped to 400ml of ammonium-carbonate water solutions of 1.5 mol/l. In addition, temperature of an ammonium-carbonate water solution was made into 20 degrees C.

Thus, the zinc oxide with a particle size of 10-25 micrometers was obtained by washing and filtering the obtained product and calcinating at 400 more degrees C.

In the example 3, the sea urchin-like particle shown in drawing 3 was obtained. The specific surface area of this zinc oxide was 43m2/g.

[0025]

(Example 1 of a comparison)

A sea urchin-like product was not able to be obtained when 200ml of zinc chloride water solutions of 0.2 mol/l and pH3 was gradually dropped to 400ml of ammonium water solutions of 1.5 mol/l.

[0026]

[Effect of the Invention]

According to this invention, a sea urchin-like zinc oxide with a large specific surface area can be obtained.

In the manufacture approach, burning temperature cannot be low, a toxic gas cannot occur, it is still higher yield and the zinc oxide of a sea urchin-like configuration can be compounded efficiently, without hardly intermingling the zinc oxide of other configurations.

[Brief Description of the Drawings]

[Drawing 1] It is the electron microscope photograph of the sea urchin-like zinc oxide compounded in the example 1.

[Drawing 2] It is the electron microscope photograph of the sea urchin-like zinc oxide compounded in the example 2.

[Drawing 3] It is the electron microscope photograph of the sea urchin-like zinc oxide compounded in the example 3.

[Translation done.]

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CLAIMS

[Claim(s)]
[Claim 1]
The zinc oxide characterized by for one end of ten or more needlelike particles gathering, and forming the sea urchin-like configuration.
[Claim 2]
It is the manufacture approach of a zinc oxide of calcinating the product which mixed the water solution (I) containing carbonate ion and/or the bicarbonate, and ammonium ion, and the water solution (II) containing zinc ion, and was obtained above 300 degrees C,
Rather than the sum total ion concentration of carbonate ion and the bicarbonate in a water solution (I), the zinc ion concentration in a water solution (II) is set up low,
pH of a water solution (II) is set to 2-7.
The manufacture approach of the sea urchin-like zinc oxide characterized by things.
[Claim 3]
The manufacture approach of the sea urchin-like zinc oxide according to claim 2 characterized by making temperature of a water solution (I) into 15-45 degrees C.

[Translation done.]